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Functional graphene oxide membrane preparation for organics/inorganic salts mixture separation aiming at advanced treatment of refractory wastewater

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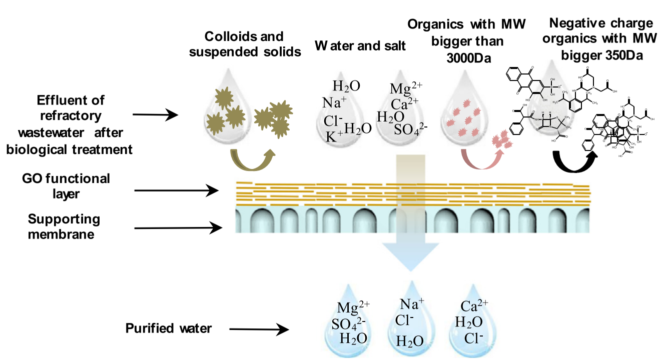
HIGHLIGHTS

- Organics/inorganic salts mixed waste produced when RO/NF treating refractory wastewater.
- Mixed waste hinder water reuse and resourcelization of inorganic salts
- Functional GO membrane was prepared, organics/inorganic salts mixed waste was avoided.
- Influence of supporting layer, GO number and preparing pressure was investigated.
- GO used was 14.4 mg/m^2, material cost was no longer limited factor for GO membrane.

GRAPHICAL ABSTRACT



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ABSTRACT

Some refractory organic matters or soluble microbial products remained in the effluents of refractory organic wastewater after biological secondary treatment and need an advanced treatment before final disposal. Graphene oxide (GO) was known to have potential to be the next generation membrane material. The functional organics/inorganic salts separation GO membrane preparation and application in wastewater advanced treatment could reduce energy or chemicals consumption and avoid organics/inorganic salts mixed concentrate waste problems after nanofiltration or reverse osmosis. In this study, we developed a novelty GO membrane aiming at advanced purification of organic matters in the secondary effluents of refractory organic wastewater and avoiding the organics/inorganic salts mixed concentrate waste problem. The influence of preparation conditions including pore size of support membrane, the number of GO layers and the applied pressure was investigated. It was found that for organics/inorganic salts mixture separation membrane preparation, the rejection and flux would achieve balance for the support membrane at a pore size of ~0.1 μm and the number of GO layers of has an optimization value (~10 layers). A higher assemble pressure (~10 bar) contributed to the acquisition of a higher rejection efficiency and lower roughness membrane. This as prepared GO membrane was applied to practical secondary effluent of a chemical synthesis pharmaceuticals wastewater. A good organic matter rejection efficiency (76%) and limited salt separation

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(<14%) was finally obtained. These results can promote the practical application of GO membrane and the resourcelized treatment of industrial wastewater.

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1. Introduction

Water security is threating nearly 80% of the world's population (Vörösmarty et al., 2000; Vörösmarty et al., 2010). One of the crises for human society is the absence of clean water (Vörösmarty et al., 2000; Vörösmarty et al., 2010), disorderly discharge of refractory organic wastewater is one of the important causes of water pollution (Ranade and Bhandari, 2014). In some developing countries, agricultural and industrial consuming too much clean fresh water, and the water leaved for the regular running of local ecosystem was far from inadequate. That caused many environmental problems (Vörösmarty et al., 2000; Vörösmarty et al., 2010). To the inland areas, wastewater reuse can be a realistic way to ease these problems.

During the production process of pharmaceuticals (Gadipelly et al., 2014), textiles (Holkar et al., 2016), petroleum refinery (Diya'uddeen et al., 2011), coal chemical industry (Ji et al., 2016; Pal and Kumar, 2013), leather (Lofrano et al., 2013), pulp-paper (Ashrafi et al., 2015), pesticide (Oller et al., 2011) and some other chemical synthesis industries (Ranade and Bhandari, 2014), a large amount of clean fresh water was consumed, and refractory organic wastewater was also produced. For economic reasons, the treatment methods of these wastewater are still dominated by biological treatment (Ashrafi et al., 2015; Diya'uddeen et al., 2011; Gadipelly et al., 2014; Holkar et al., 2016; Ji et al., 2016; Lofrano et al., 2013; Oller et al., 2011; Pal and Kumar, 2013; Ranade and Bhandari, 2014). However, limited by hydraulic retention time, biodegradable potential, introduction of microbial products and the huge fluctuations of in inlet wastewater quality and emission volume, a certain amount of organic matters often leaved in the effluent after biological treatment (Laspidou and Rittmann, 2002; Oller et al., 2011; Sheng et al., 2010; Shon et al., 2006; Xie et al., 2016). These organic matters often include the original refractory components (Oller et al., 2011), bio-converted self-polymerized products and soluble microbial products (Azami et al., 2012; Laspidou and Rittmann, 2002; Sheng et al., 2010). In the follow-up disinfection process, some of them tend to be converted to disinfection by-products (Doederer et al., 2014). These disinfection by-products (Doederer et al., 2014) and residual refractory organic matters discharged to the environment will bring water pollution problems and other environmental risks (Jiang et al., 2013). So, these residual refractory organic matters often require advanced treatment (tertiary treatment) (Oller et al., 2011).

The existing popular advanced treatment technology includes advanced oxidation [19, 20] and membrane filtration (Li et al., 2011) process. The advanced oxidation technology is usually effective, but tends to have high consumption to chemicals (Deng and Englehardt, 2006; Oturan and Aaron, 2014) or energy (Ternes et al., 2003). Other problems can be chemical sludge from Fenton process (Neyens and Baeyens, 2003), residual ozone from ozone oxidation process causing air pollution (Altmann et al., 2014) and other toxic by-products problems (Pignatello et al., 2006; Wang et al., 2013). All of these limit the application of these technologies. Nanofiltration and reverse osmosis (NF/RO) technology can achieve water reuse, but tend to have a high energy consumption (Werber et al., 2016) and serious membrane fouling (Zhao and Yu, 2014) problems. Polyamides (PA) and other organic membrane have limited antioxidant capacity, making the fouling control and membrane cleaning process difficult (Werber et al., 2016). These hinder the application of membrane filtration technology in wastewater advanced treatment process (Werber et al., 2016). In addition, the concentrated waste generate after NF/RO (Dialynas et al., 2008; Van der Bruggen et al., 2003). As the inorganic salts content of refractory wastewater is often high (Perez-Gonzalez et al., 2012), the concentrated waste tended be an organics/inorganic salts mixed state that usually exceeded the direct emission standards (Mickley, 2001). Organics/inorganic salts mixed concentrate waste often led to the follow-up concentrated water evaporation process heavy fouling (Rautenbach and Mellis, 1995) and cause the crystallize of inorganic salt difficult to carry on (Perez-Gonzalez et al., 2012) that due to the coordination effect of organic matter to inorganic (Song et al., 2014). These affect the resourcelization of salts and the final mineralization of organic matters (Pignatello et al., 2006). So the organic and inorganic matters should be separated, treated or resource utilized respectively (Rautenbach and Mellis, 1995).

Graphene oxide (GO) was known to have potential to be the next generation of membrane material (2015; Mi, 2014; You et al., 2015; Zhao et al., 2015). It showed good chemical stability (Choi et al., 2013; Dreyer et al., 2010), fine rejection efficiency (Joshi et al., 2014; Li et al., 2013; Morelos-Gomez et al., 2017; Radha et al., 2016; Sun et al., 2016) and have potential to be prepared in a relative low price (Hummers and Offeman, 1958; Joshi et al., 2015; Marcano et al., 2010). GO and GO like materials also shows a wide range of applications in other areas of the environment (Li et al., 2017; Yao et al., 2017a; Yao et al., 2017b; Yin et al., 2017). A great amount of attention has been paid to obtain desalination GO membrane (Cheng et al., 2016; McGuinness et al., 2015; Radha et al., 2016; Werber et al., 2016; You et al., 2015) that have high flux and rejection efficiency (Abraham et al., 2017; Chen et al., 2017; Kidambi et al., 2017; Morelos-Gomez et al., 2017; Wang et al., 2017) or solve the problem GO layer swelling in aqueous solution (Abraham et al., 2017; Zheng et al., 2017). The research related to organics/inorganic salts separation GO membrane preparation and application was still lacking. Since the bio-converted self-polymerized organic products and soluble microbial products of refractory wastewater secondary effluent tend to have high molecular weight (Shon et al., 2006; Xie et al., 2012; Yang et al., 2013), it is possible to separate these organic matters from salts and water. If GO membrane can be prepared as the targeted functional membrane and used in advanced treatment of refractory wastewater, it could contribute to solve the high chemical (Deng and Englehardt, 2006; Oturan and Aaron, 2014) or energy (Ternes et al., 2003; Werber et al., 2016) consumption problem, and the organics/inorganic salts mixed concentrated waste problem (Perez-Gonzalez et al., 2012; Rautenbach and Mellis, 1995). These widely existed in the current advanced treatment process to refractory wastewater. However, the GO membrane preparation study aiming at refractory wastewater advanced treatment was relatively fewer (Lee et al., 2013). The influence of supporting membrane pore size, GO layer number (Kim et al., 2013) and preparing pressure (Tang et al., 2014; Tsou et al., 2015) on the organics/inorganic salts separation property and other characteristics of GO membrane was not explored enough.

In this study, efforts were made to prepare a GO membrane that capable to organic matters and salts separation, avoiding organics/inorganic salts mixed concentrate waste problem and promote the resourcelization of inorganic concentrate waste during membrane filtration. The influence of GO layer number, support layer material and pore size, and GO nanosheets assemble pressure to the flux, rejection and robustness of prepared membrane was investigated. The optimized GO membrane had a COD rejection efficiency of 76%, and salts rejection < 14% to a real secondary effluent of refractory pharmaceutical wastewater. Results of this study opened a new approach for GO membrane applying to the advanced purification of wastewater.

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